6 2 2002 075

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: 0 360 307 A1

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: 89201821.9

(22) Date of filing: 07.07.89

(51) Int. Cl.5: C04B 37/00, C04B 35/10, H01J 61/36

(30) Priority: 07.09.88 HU 461488

(43) Date of publication of application: 28.03.90 Bulletin 90/13

(84) Designated Contracting States: BE DE GB NL

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(54) **Soldering enamel for preparing an end seal of a ceramic discharge envelope of a discharge lamp.**

(57) The soldering enamel is composed of a basic enamel consisting of aluminium oxide and a rare earth metal oxide and/or an alkali earth metal oxide and, in the given case, some other metallic or metalloid oxide, and of a ceramic filling material. The filling material is a zirconium-rare earth oxide of perovskite crystal structure. The quantity of the filling material amounts up to 5 mass% referred to the overall quantity of enamel.

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SOLDERING ENAMEL FOR PREPARING AN END SEAL OF A CERAMIC DISCHARGE ENVELOPE OF A DISCHARGE LAMP

OBJECT OF THE INVENTION

The present invention refers to a soldering enamel suitable for preparing the end seal of a ceramic envelope of a discharge lamp.

In the course of manufacturing different kinds of discharge lamps and especially the high-pressure sodium vapour (HPS) lamps the step of sealing of the ceramic discharge envelope is the critical technological operation. In preparing the sealing of said discharge envelopes at least the current inlets have to be fixed in a pressure-tight manner into the ceramic envelope but in many cases also the discharge envelope itself requires to be provided with a ceramic sealing element of ceramic plug also attached in a pressure-tight way to the adjacent part of the ceramic discharge envelope, this part being of tubular shape in most cases. For this purpose of fixing so-called enamel solders of different kinds are used. This is usually done so that from the enamel of suitable composition, a suitably matching element of proper shape, e.g. an enamel ring is formed, that is then put to the required place and by heating the assembly thus fitted together, the enamel is melted.

The molten enamel wets the parts to be soldered together and flows into the gaps between them. After cooling the assembly, the enamel solidifies, and a pressure-tight bond between the parts to be soldered together is ensured by this solid enamel. The quality of the bond formed this way is highly influenced by the phase composition of the solidified enamel. While solidifying, the enamels tend to take part in processes resulting in a lower or higher percentage of a glass phase. When in the solidified enamel a relatively large percentage of the glass phase is present, then it is susceptible to cracking and also its sodium resistance will be poor. Even if formation of the glassy phase is avoided, it will still be difficult to obtain a solidified enamel of satisfactory texture, i.e. of one consisting of the required phases. This is however, of great importance, since formation of certain phases must by all means be avoided, namely of those which show a microporous zeolitelike structure, these phases being inclined to get bound to gases formed and also their sodium resistance is poor - and also the development of those phases should be prevented which are subject to phase transformation, while the discharge lamp is heated to service temperature. These unwanted phases are likely to appear, if the formation rate of solidification nuclei, around which the desired phases develop, is unsatisfactory and if the enamel is prone to undercooling.

To obtain a solidified enamel of the required fine structure and consisting of the required phases, it is expedient to admix to the base enamel composition from some dope capable of promoting the nucleation process, the dope consisting of a melt not prone to undercooling. Namely in that case the speed of the nucleation process will not influence the quality of enamel. Under the effect of these implanted crystal grains the solidification process sets in simultaneously within the mass of enamel still in liquid phase, so as if the matter were "hogged away" from other undesirable phases, and on the other hand, by locally modifying the composition of the melt, solidification of undoped, but desirable phases also sets in. These crystal grains remain partly in solid state during the soldering-in process. (This obviously also depends on the soaking time.) The structure of said grains adjusts itself to the formation of the required phase, while also their grain size is uniform, remains in conformity with the fine structure.

The method of using nuclei forming dopes in the technical art of the present application is already known. So, in the U.S. patent specification No. 4 585 972, admixture to the enamel of 2 to 5 mass% phosphorous pentoxide, titanium dioxide, zirconium dioxide or chromium trioxide individually or in any arbitrary combination is proposed.

It is a known method as well to add to the enamel a ceramic filling having the additional property of expediting uniform solidification of the enamel, as stated in the European patent application No. EP 237 103. In that specification, admixture of 20 to 50 percent of a filling consisting of any one or any combination of the following substances is proposed: aluminium dioxide, thorium dioxide, uranium dioxide and, advantageously, hafnium dioxide. The use of a such high rate of the filling material is disadvantageous because it has the adverse effect of rendering the enamel thixotropic, impending easy intrusion of the enamel into the gaps to be soldered.

SUMMARY OF THE INVENTION

The present invention is directed to creating an improved soldering enamel showing low thixotropy, the required fine structure and phase composition in spite of applying a filling material.

The present invention is based on the recognition that the filling material should be also the additive improving the nucleation process. This re-

of grains smaller than the specified lower limit is undesirable, since such grains will melt into the base enamel during soldering and will be impended in exerting its desired effect as filling material. Particles of grain sizes larger than the specified upper limit, on the one hand, encumber the preparation of the enamel ring and on the other hand, will fail to provide a sufficient number of nuclei in the microcrystal structure of the given volume of the basic enamel.

Example 2

To a moulding powder consisting of a ground powder of 33.2 mass% Al_2O_3, 52.6 mass% $CaCO_3$, 3.7 mass% Y_2O_3 and 10.5 mass% $SrCO_3$
mixed together with a few mass% of some organic binding material, e.g. PVA (polyvinyl-alcohol), then 0.5 mass% the material prepared according to Example 1 is admixed (instead of carbonates and oxides, oxalates and nitrates can equally be used, provided their molar quantities correspond to those specified above). In that case the organic binding material can be omitted. From the moulding powder thus prepared a ring (or circlet) was pressed in an otherwise known way, then this moulded ring was heated up to 1240-1260 °C slowly, through several hours and kept at the temperature for a period of 1 hour. The glowed enamel ring can be used (preferably at once) for the sealing of ceramic discharge envelopes of sodium lamps. The assembled fitting can then be soldered together at 1450°C. The heating-up time depends on the design of the oven and lamp body.

The duration of holding time is 1 to 2 minutes. The rate of cooling is determined by that of the oven.

Example 3

A mixture consisting of
331.4g of Al_2O_3, 84.5 g of Sc_2O_3,
53.3g of Y_2O_3 and 243.1 g La_2O_3
was ground and melted and, after cooling, grinding was carried on until the grain size was reduced to 3 to 5 μm. To this powder 1.5 g of the material prepared according to Example 1 and an organic binding material (e.g. PVA) facilitating the process of moulding were admixed.

A ring was moulded from the above mixture of powders and by slow heating through several hours its temperature was raised to 1450°-1500 °C and kept there for 30 minutes.

The enamel ring can be used for fixing the ceramic discharge envelope of sodium lamps by soldering, in a way similar to those described in the preceding examples with the difference that the soldering temperature is max. 1670 °C.

Claims

1. Soldering enamel for preparing an end seal of a ceramic discharge envelope of a high-pressure discharge lamp, comprising a basic enamel composed of aluminium oxide and a rare earth oxide and/or alkaline earth oxide and, in the given case, of some other metallic or metalloid oxide, and containing a ceramic filling material, **characterized by** that the ceramic filling material is a zirconium-rare earth oxide of perovskite crystal structure and the amount of the filling material is at most 5 mass% of the overall mass of the basic enamel, for facilitating the nucleation process and forming a mixture of low thixotropy during soldering.

2. Soldering enamel as set forth in claim 1, **characterized by** containing at most 1 mass% of the zirconium-rare earth oxide perovskite.

3. Soldering enamel as set forth in claim 1 or 2, **characterized by** containing 0.1 to 0.9 mass% yttrium-zirconium oxide of perovskite crystal structure having a ceramic filling material in form of preferred composition expressed by the general formula $Y_{0.29377}Zr_{1.32468}O_3$.

European Patent Office

EUROPEAN SEARCH REPORT

Application number

EP 89201821.9

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,A	EP - A1 - 0 237 103 (N.V.PHILIPS' GLOEILAMPEN-FABRIEKEN) * Claims * ----	1	C 04 B 37/00 C 04 B 35/10 H 01 J 61/36
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 04 B H 01 J B 23 K

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	11-12-1989	BECK

CATEGORY OF CITED DOCUMENTS

X particularly relevant if taken alone
Y particularly relevant if combined with another document of the same category
A technological background
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T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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